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Chevron Phillips Chemical Company LP

201-15220

I U C L I D

Data Set

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Existing Chemical	:	ID: 68201-32-1
CAS No.	:	68201-32-1
EINECS Name	:	Asphalt, sulfonated, sodium salt
EINECS No.	:	269-212-0

Producer Related Part

Company	:	Chevron Phillips Chemical Company LP
Creation date	:	09.01.2004

Substance Related Part

Company	:	Chevron Phillips Chemical Company LP
Creation date	:	09.01.2004
Memo	:	
Printing date	:	30.01.2004
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Flags (profile)	:	Flags: without flag, confidential, non confidential, WGK (DE), TA-Luft (DE), Material Safety Dataset, Risk Assessment, Directive 67/548/EEC, SIDS

1.0.1 OECD AND COMPANY INFORMATION

Type	:	other
Name	:	Chevron Phillips Chemical Company LP
Partner	:	
Date	:	
Street	:	10001 Six Pines Drive
Town	:	77380 The Woodlands, TX
Country	:	United States
Phone	:	
Telefax	:	
Telex	:	
Cedex	:	

09.01.2004

2.1 MELTING POINT

2.2 BOILING POINT

2.4 VAPOUR PRESSURE

2.5 PARTITION COEFFICIENT

Log pow	:	< 0 - 6.2 at 22° C
Method		OECD Guide-line 117 "Partition Coefficient (n-octanol/water), HPLC Method"
Year	:	1997
GLP	:	yes
Test substance	:	other TS

Remark : The following interpretation of the results were provided by Ambiorn Hanstveil (TNO Nutrition and Food Research Institute, Toxicology Division) to the Drilling Specialties Company:

- Four components with a log Pow </= 3, i.e. peak numbers 1 to 4, are considered to have no potential for bioaccumulation.
- One component with a log Pow = 3.2, i.e. peak number 5, is a limit case. Depending on its molecular weight, it will have a limited potential for bioaccumulation or none at all.
- Two components with a log Pow > 6.2, i.e. peak numbers 7 and 8, have extreme long retention times compared to the reference substances. These peaks are therefore considered to represent components with very high log Pow values, that have no potential for bioaccumulation.

Result : The partition coefficient (n-octanol/water) range determined for Soltex Shale Inhibitor by HPLC was:

Range log Pow: < 0, 1.1, 3.2, and > 6.2

It was noted that Soltex Shale Inhibitor did not completely dissolve in methanol. From the observation of eight major peaks in HPLC chromatograms, the partition coefficient (log Pow) for the fraction of Soltex Shale Inhibitor soluble in methanol, was calculated to range from < 0 to > 6.2, i.e. lower than zero and higher than the highest partition coefficient of the reference components, which in this study was 4,4'-DDT. In addition to the major eight peaks, minor peaks could be observed in the chromatogram of the test substance after elution of the last reference component. The partition coefficients for these peaks can also be regarded as > 6.2.

RESULTS FOR SOLTEX SHALE INHIBITOR:
Results are presented in the following format: peak / tR (min) / k / log k / calculated log Pow:

peak 1 / 1.27 / -0.56 / n.a. / < 0
peak 2 / 1.46 / -0.49 / n.a. / < 0
peak 3 / 1.98 / -0.31 / n.a. / < 0
peak 4 / 2.77 / -0.03 / n.a. / < 0
peak 5 / 3.04 / 0.06 / -1.201 / 1.1

peak 6 / 4.21 / 0.47 / -0.326 / 3.2
peak 7 / 164.69 / 56.58 / n.a. / > 6.2
peak 8 / 222.30 / 76.73 / n.a. / > 6.2

Explanation:
k: capacity factor = (tR - t0) / t0
tR: retention time of the reference or test substance
t0: dead-time (i.e. retention time of formamide)
n.a.: not applicable, not within range of reference substances

RESULTS FOR REFERENCE SUBSTANCES:
Results are presented in the following format: reference material / tR (min) / k / log k / log Pow / log Pow back-calculated:

phenol / 3.03 / 0.06 / -1.23 / 1.5 / 1.1
phenol / 3.05 / 0.07 / -1.18 / 1.5 / 1.2
toluene / 4.20 / 0.47 / -0.33 / 2.7 / 3.2
toluene / 4.19 / 0.47 / -0.33 / 2.7 / 3.2
naphthalene / 4.98 / 0.74 / -0.13 / 3.6 / 3.7
naphthalene / 4.92 / 0.72 / -0.14 / 3.6 / 3.7
biphenyl / 6.33 / 1.21 / 0.08 / 4.0 / 4.2
biphenyl / 6.11 / 1.14 / 0.06 / 4.0 / 4.1
phenanthrene / 10.01 / 2.50 / 0.40 / 4.5 / 5.0
phenanthrene / 9.50 / 2.32 / 0.37 / 4.5 / 4.9
4,4'-DDT / 14.69 / 4.14 / 0.62 / 6.2 / 5.5
4,4'-DDT / 13.47 / 3.71 / 0.57 / 6.2 / 5.4

Explanation:
k: capacity factor = (tR - t0) / to
tR: retention time of the reference or test substance
to: dead-time i.e. retention time of formamide: 2.86 minutes (average of 2.88 and 2.84 minutes)

Source : Phillips Petroleum Company, Determination of the Partition Coefficient (n-octanol/water), HPLC method, of Soltex Shale Inhibitor. Study performed by BCO Analytical Services B.V, Breda, The Netherlands for Drilling Specialties Company, Bartlesville, Oklahoma.

Test condition : TEST AND REFERENCE MATERIALS
- Test substance was a product sample of Soltex Shale Inhibitor (Drilling Specialties Company, Bartlesville, Oklahoma).
- Chemical composition: sulphonated asphaltenes
- (Hot) water solubility: high (determined by Soxhlet extraction with water)
- References substances were:
----phenol, 99.5%, CASN 108-95-2
----toluene, 99%, CASN 108-88-3
----naphthalene, 100%, CASN 91-20-3
----biphenyl, 99%, CASN 92-52-4
----phenanthrene, 98%, CASN 85-01-8
----4,4'-DDT, 99%, CASN 50-29-3
- Preparation of test solutions: A solution of Soltex Shale Inhibitor was prepared by accurately weighing 11 mg and dissolving in 100 ml methanol (99.5%, CASN 67-56-1). The fraction soluble in methanol was further used in the study. The test solution was used directly for preparation of UV/VIS spectrum and concentrated ten times (by evaporation) for HPLC analysis.
- Preparation of reference substance and formamide solutions (a mixture was prepared form the following solutions by combining 200 ul of the solutions of phenol, toluene, naphthalene and 4,4'-DDT and 20 ul of the solutions of biphenyl and phenanthrene with 100 ul of the formamide solution):
----phenol: 11 mg dissolved in 10 ml methanol
----toluene: 149 mg dissolved in 10 ml methanol

----naphthalene: 10 mg dissolved in 10 ml methanol
----biphenyl: 11 mg dissolved in 1.0 ml methanol
----phenanthrene: 10 mg dissolved in 10 ml methanol
----4,4'-DDT: 9 mg dissolved in 10 ml methanol
----formamide: 110 mg dissolved in 10 ml methanol

EQUIPMENT AND REAGENTS
- The HPLC was equipped with a Vydac 201 TPB (C18, 25 cm id. 4.6 mm) column.
- Elution: isocratic
- Mobile phase: methanol / Suprapur water 75:25 (v/v)
- Detector: UV 210 or 254 nm
- Flow rate: 1.0 ml/minute
- Temperature: 22 +/- 0.2 deg C
- Injection volume: 20 ul

METHOD OF ANALYSIS
- Determination of dead time: determined from the duplicate measurements of retention time for formamide.
- References and calibration: A calibration graph was prepared for the references in order to correlate the measured capacity factor k with the Pow of the test substance. The mixture of the reference substances was injected in duplicate onto the HPLC column and the resulting retention times were measured. The calibration curve was prepared by plotting log k versus log Pow for the reference compounds.
- Log Pow values of the reference substances were:
----phenol = 1.5
----toluene = 2.7
----naphthalene = 3.6
----biphenyl = 4.0
----phenanthrene = 4.5
----4,4'-DDT = 6.2

CONDUCT OF THE TEST
- HPLC analysis of the test substance was performed and the range of retention times of the detectable components was determined. In addition, the retention times of peaks that could be distinguished within this range were measured.
- The applicability of the use of the wavelengths (210 or 254 nm) of the UV detector was verified by recording the UV/VIS absorption spectrum of the test solution of 0.11 g/l Soltex Shale Inhibitor in methanol (using blank methanol as reference).
- The spectrum was recorded using a Perkin-Elmer lambda 2 UV/VIS spectrometer and quartz cuvette (pathlength 1 cm).

CALCULATIONS
- Calibration curves were prepared by linear regression analysis with spreadsheets in Lotus 1-2-3 version 2.2.
- The HPLC analysis of the test substance resolved in a band of analytical signals (some clearly visible as peaks) on the HPLC chromatograms. From the first and last detectable signal of the test substance, the upper and lower limits of log Pow were determined. In addition, the log Pow was calculated for the clearly distinguishable peaks.
- Results (partition coefficient of the test substances) of peaks within the range of retention times of reference substances, were calculated by interpolation of the calculated capacity factor k on the calibration curve.
- When peaks were outside the range of retention times observed for the reference substances, the log Pow values were set at either below zero or greater than 6.2.

Test substance : Asphalt, sulfonated, sodium salt, CAS Number 68201-32-1. XXXXXXXX XXXXXXX. Trade Name: Soltex Shale Inhibitor.

Reliability : (1) valid without restriction
Flag : Critical study for SIDS endpoint
30.01.2004 (1)

Log pow : < 0 at ° C
Method OECD Guide-line 117 "Partition Coefficient (n-octanol/water), HPLC Method"

Year : 2003
GLP : yes
Test substance : other TS

Result : Three peaks were detected with UV and none with RI. The log Pow values were all < 0.0.

These results show all peaks have log Pow values less than 3.0 and are consistent with a material which has little tendency to accumulate in the environment.

Results are for the water soluble portion of the sample only and are provided in the following format:
Peak / Retention time (mins) Run 1 / Retention time (mins) Run 2 / Peak Area (%) Run 1 / Peak Area (%) Run 2 / k Run 1 / k Run 2 / log k Run 1 / log k Run 2 / Log Pow Run 1 / Log Pow Run 2

Peak 1 / 2.044 / 2.247 / 85.38 / 86.11 / -0.25 / -0.18 / * / * / * / *
Peak 2 / 2.336 / 2.344 / 10.40 / 9.89 / -0.15 / -0.15 / * / * / * / *
Peak 3 / 2.605 / 2.614 / 4.22 / 4.01 / -0.05 / -0.05 / * / * / * / *

* Unable to calculate as k is negative (tR < t0)
t0 = 2.744

CALIBRATION DATA:
Results are presented in the following format: Reference material / Retention time (mins) / k / log k / log Pow from the literature:

Benzene / 4.983 / 0.82 / -0.09 / 2.1
Toluene / 6.385 / 1.33 / 0.12 / 2.7
Ethyl benzene / 8.125 / 1.96 / 0.29 / 3.2
Propyl benzene / 11.168 / 3.07 / 0.49 / 3.7
Butyl benzene / 16.067 / 4.86 / 0.69 / 4.6
DDT / 35.547 / 11.96 / 1.08 / 6.2

t0 = 2.744

Source : Phillips Petroleum Company, The Bioaccumulation Potential of Sulphonated Asphalt Additive - Report for Drilling Specialties Company. Study performed by Chemex Environmental International Limited, Cambridge, England for Drilling Specialties Company, Bartlesville, Oklahoma.

Test condition : TEST SUBSTANCE: Sulphonated Asphalt Additive supplied by Drilling Specialties Company, purity not known.

REFERENCE SUBSTANCES:
- Benzene: log Pow (from literature) 2.1, 99.7%, 0.0016 ul injected.
- Toluene: log Pow (from literature) 2.7, 99.95%, 0.0016 ul injected.
- Ethyl benzene: log Pow (from literature) 3.2, 99.0%, 0.0016 ul injected.
- Propyl benzene: log Pow (from literature) 3.7, 98.0%, 0.0016 ul injected.
- Butyl benzene: log Pow (from literature) 4.6, 99+%, 0.0016 ul injected.
- DDT: log Pow (from literature) 6.2, 98.0%, 6.86 ug injected.

- Thiourea: 99.0%, 0.15 ug injected.

SAMPLE PREPARATION
- 0.1 g of test material was dispersed in 10 ml of pH 8 aqueous buffer and syringe filtered (0.45 um) to remove un-dissolved sample. 7.5 ml of methanol was added to 2.5 ml of the filtrate and this was injected in duplicate (0.025 g in 10 ml). The quantities injected were 0.05 mg.

INSTRUMENTATION
- Chromatography System: Perkin Elmer Quaternary System
- HPLC gradient pump: Perkin Elmer Series 200
- UV detector: Perkin Elmer 785A UV/VIS @ 210 nm (1.0V/AU)
- RI detector: Perkin Elmer LC-25
- Interface box: 900 Series and 600 Link Series
- Software: PE Nelson Turbochrom Workstation
- Analyical column: Hypersil, 5 um, C18, 250 by 4.6 mm

CONDITIONS
- Mobile phase: 75:25 methanol: 0.02M phosphate buffer (pH 8.0)
- Flow rate: 1 ml/min
- Injection volume: 20 ul (standard) and 20 ul (sample and blank)
- The system dead time (t0) is the average retention time of a non-retained material. the dead time is taken as the retention time of the thiourea peak (or the solvent front when the thiourea and solvent coelute).

Test substance : Asphalt, sulfonated, sodium salt, CAS Number 68201-32-1. XXXXXXXXX XXXXX.

Reliability : (1) valid without restriction
Flag : Critical study for SIDS endpoint
30.01.2004 (3)

2.6.1 WATER SOLUBILITY

3.1.1 PHOTODEGRADATION

3.1.2 STABILITY IN WATER

3.3.1 TRANSPORT BETWEEN ENVIRONMENTAL COMPARTMENTS

Type	:	fugacity model level III
Media	:	other: air-water-soil-sediment
Air (level I)	:	
Water (level I)	:	
Soil (level I)	:	
Biota (level II / III)	:	
Soil (level II / III)	:	
Method	:	other: EPIWIN v 3.10
Year	:	2004

Method : Used EPIWIN v 3.10. The following physical properties were used as the model input parameters:

Chem Name: Three representative structures, C26 H45 O3 S1 Na1; C26 H43 O9 S3 Na3; and C40 H61 O15 S5 Na5
Molecular Wt: 460.7; 664.78; 1057.2
Henry's LC (atm-m3/mole): 6.07E-7; 1.79E-20; 9.33E-34
Vapor Press (mm Hg): 6.02E-18; 3.9E-23; 1.75E-33
Liquid VP (mm Hg): 5.51E-15; 6.36E-20; 2.86E-30
Melting Pt (deg C): 324; 350; 350
Log Kow: 6.78; 2.32; 4.05
Soil Koc: 2.47E+6; 85.7; 4.6E+3

Result : Results are provided in the following format:
Compartment / 100% to Air/ 100% to Water / 100% to Soil/ Equally to Each Compartment

(C26 H45 O3 S Na)
Air / 5.33% / 0.000131% / 0.00% / 0.444%
Water / 2.47% / 12.3% / 0.00161% / 8.2%
Soil / 74.7% / 0.00183% / 100% / 33.1%
Sediment / 17.5% / 87.7% / 0.0114% / 58.2%

Persistence when distributed equally to each compartment = 643 hr
(Emissions [kg/hr] = 1000 to air, 1000 to water, 1000 to soil, and 0 to sediment).

(C26 H43 O9 S3 Na3)
Air / 0.0055% / 0.00% / 0.00% / 0.35%
Water / 10.3% / 99.5% / 5.66% / 31.5%
Soil / 89.6% / 0.00% / 94.3% / 68.0%
Sediment / 0.0557% / 0.537% / 0.0306% / 0.17%

Persistence when distributed equally to each compartment = 707 hr
(Emissions [kg/hr] = 1000 to air, 1000 to water, 1000 to soil, and 0 to sediment).

(C40 H61 O15 S5 Na5)
Air / 0.00% / 0.00% / 0.00% / 0.00246%
Water / 3.55% / 82.3% / 0.156% / 15.3%
Soil / 95.7% / 0.00% / 99.8% / 81.4%
Sediment / 0.763% / 17.7% / 0.0334% / 3.29%

Persistence when distributed equally to each compartment = 1.6E+3 hr (Emissions [kg/hr] = 1000 to air, 1000 to water, 1000 to soil, and 0 to sediment).

Source : EPI Suite v 3.10.

Test substance : Three representative structures: C26 H45 O3 S1 Na1; C26 H43 O9 S3 Na3; and C40 H61 O15 S5 Na5

Reliability : (2) valid with restrictions
Flag : Critical study for SIDS endpoint
30.01.2004 (16)

3.5 BIODEGRADATION

Type : aerobic
Inoculum :
Concentration : 3 mg/l related to Test substance

Contact time : 56 day
Degradation : 0 - 3 % after 56 day
Result : under test conditions no biodegradation observed
Deg. Product :
Method : other: EC Guideline "Biotic degradation in seawater: Closed Bottle Method"
Year : 1993
GLP : yes
Test substance : other TS

Result : No oxygen consumption was found which could be attributed to the biological degradation of Soltex.

The results showed the expected rapid degradation of acetate (complete within seven days). The measured oxygen consumption of acetate was 0.66 mg O2/mg after seven days and compared well with the theoretical oxygen demand of 0.68 mg O2/mg.

The calculated oxygen consumption of acetate in the presence of the test substance was slightly lower than found with acetate alone, indicating that Soltex had a slightly inhibiting effect on the acetate degradation.

The oxygen consumption due to the acetate in presence of the 3.5% bentonite slurry was similar to that of acetate and Soltex, confirming that the effect recorded above was probably caused by the bentonite slurry.

Soltex was not degraded in the presence of acetate.

Source : Phillips Petroleum Company, The Biodegradability of the Product 3.5% Bentonite Slurry with 1% Soltex (262:100-2) in Seawater According to a Proposed EC Test Guideline (Closed Bottle Test). Study performed by TNO Environmental and Energy Research, Delft, The Netherlands for Drilling Specialties Company, Bartlesville, Oklahoma.

Test condition : TEST SUBSTANCE
- 3.5% bentonite slurry with 1% Soltex (262:100-2), a dark brown liquid.

NATURAL SEAWATER
- A sample of natural seawater was taken from the Eastern Scheldt (Jacobahaven) on August 28, 1991.
- Water temperature was 18.5 deg C and the salinity was 33.3%.
- The sample was taken 2.5 meters above the bottom about one hour after low tide and was aerated until the test started.
- TOC of the seawater was found to be <0.8 mg C/L.
- Total chlorophyll and phaeophytine content: 4.30 - 4.95 mg/m3.
- Content of NO3, NH4+, and PO4-3 was < 15, 0.16, and <0.10 mg/L respectively.
- 1 ml of each of the following nutrient stock solutions were added per litre of natural seawter prior to use in order to prevent nutrient limitation:
----Stock solution a per 1000 ml milli-Q-water: 8.50 g KH2PO4, 21.75 g K2HPO4, 50.14 g Na2HPO4.7H2O, 1.70 g HN4Cl
----Stock solution b per 1000 ml milli-Q-water: 22.5 g MgSO4.7H2O
----Stock solution c per 1000 ml milli-Q-water: 36.4 g CaCl2.2H2O
----Stock solution d per 1000 ml milli-Q-water: 0.15 g FeCl3

TEST METHOD
- A concentration of 2 mg test substance per litre usually allows the determination of 95% degradation. However, on the basis of TNO's experience in testing this chemical substance in seawater, one high concentration was tested.
- A test substance concentration equivalent to about 3 mg/L of Soltex was used.
- A test concentration of 301 mg/L (262:100-2, corresponding to 3.0 mg/L Soltex) was prepared by adding 1.8038 g of test substance to 6.0 litre of natural seawater.
- In order to check the toxicity of the test substance, a test concentration of 299.5 mg/L was prepared by adding 1.2580 g of test substance to 4.2 litre of natural seawater containing 4.0 mg/L sodium acetate.
- In order to check the bacterial activity of the seawater itself additional bottles were prepared containing only 4 mg/L soidum acetate as carbon source.
- Each test solution was distributed over thirteen bottles. In addition, nineteen bottles were prepared with natural seawater only to serve as control for the oxygen consumption by the seawater itself.
- Triplicate BOD bottles were prepared for each treatment. The initial oxygen concentration was measured with an oxygen electrode in one bottle of each treatment.
- The other bottles were then closed and incubated at about 20 deg C in the dark.
- The O2 concentrations were measured again after 7, 14, 21, and 28 days (all treatments) and 42 and 56 days (reference and test substance). A separate set of bottles was sacrificed for each measurement.

CALCULATION OF RESULTS
- The oxygen demand in each test bottle after 7, 14, 21, 28, 42 and 56 days was calculated by subtracting the oxygen concentration measured at that time from that measured at the start of the test.
- The Biochemical Oxygen Demand (BOD) due to the test or control substances at each time was calculated (in mg O2/L) by subtracting the oxygen demand in the relevant inoculum control or other control bottle from that in the bottle under consideration; these crude values were then converted to values per mg substance.
- The degradation was expressed as BOD as percentage of the COD of the test substance.

Test substance : 3.5% bentonite slurry with 1% Soltex (Asphalt, sulfonated, sodium salt, CAS Number 68201-32-1. (XXXXXXXXXXXX).

Reliability	:	(1) valid without restriction
Flag	:	Critical study for SIDS endpoint
30.01.2004		(12)
Type	:	aerobic
Inoculum	:	
Concentration	:	3.8mg/l related to Test substance
Contact time	:	28 day
Degradation	:	3 - 6 % after 28 day
Result	:	other: low biodegradability in seawater.
Deg. Product	:	
Method	:	other: EC Guideline "Biotic degradation in seawater: Closed Bottle Method"
Year	:	1991
GLP	:	yes
Test substance	:	other TS

Result :

The oxygen consumption due to the test substance was low, representing at most 3-6% of the Chemical Oxygen Demand (COD). It was concluded that Soltex Shale Inhibitor has a low biodegradability in seawater.

The results showed the expected rapid degradation of the acetate control substance (complete within 14 days). The measured oxygen consumption of acetate was 0.66 mg O2/mg after 14 days and compared well with the theoretical oxygen demand (TOD) of 0.68 mg O2/mg.

Source :

Phillips Petroleum Company, The Biodegradability of the Product Soltex Shale Inhibitor in Seawater According to a Proposed EC Test Guideline (Closed Bottle Test). Study performed by TNO Environmental and Energy Research, Delft, The Netherlands for Drilling Specialties Company, Bartlesville, Oklahoma.

Test condition :

TEST SUBSTANCE
- Soltex Shale Inhibitor, batch no. 90-7101, a dark brown to black granular material.

NATURAL SEAWATER
- A sample of natural seawater was taken from the Eastern Scheldt (Jacobahaven) on April 9, 1991.
- Water temperature was 9 deg C and the salinity was 3.26%.
- The sample was taken 2.5 meters above the bottom about half an hour before high tide and was aerated until the test started.
- Total chlorophyll and phaeophytine content: 4.8 - 5.9 mg/m3.
- Content of NO3, NH4+, and PO4-3 was 2.4, 0.06, and 0.08 mg/L respectively.
- 27 ml of each of the following nutrient stock solutions were added to 27 litres of natural seawter prior to use in order to prevent nutrient limitation:
----Stock solution a per 1000 ml milli-Q-water: 3.50 g KH2PO4, 21.75 g K2HPO4, 50.14 g Na2HPO4.7H2O, 1.70 g HN4Cl
----Stock solution b per 1000 ml milli-Q-water: 22.5 g MgSO4.7H2O
----Stock solution c per 1000 ml milli-Q-water: 36.4 g CaCl2.3H2O
----Stock solution d per 1000 ml milli-Q-water: 0.15 g FeCl3

TEST METHOD
- A concentration of 2 mg test substance per litre usually allows the determination of 95% degradation. It was not possible to disperse the test substance in water or an organic solvent and a higher concentration had to be tested.
- About 1 mg dry test substance was added to each bottle of about 295 ml (the volume of the individual bottles varied between 294.9 and 295.3 ml) resulting in a mean test substance "concentration" of about 4 mg/L.
- Two series of 12 bottles were prepared, one containing the test substance

alone, and one toxicity control series containing 4 gm/L sodium acetate in addition to the test substance.
- Two bottles without test substance were prepared for determination of the initial oxygen concentration.
- In order to check the bacterial activity of the seawater itself, 13 additional bottles were prepared containing only 4 mg/L sodium acetate as carbon source.
- In addition, 13 bottles with seawater without any additions were prepared to serve as a control series for the oxygen consumption by the seawater itself.
- After the oxygen concentration had been determined in one bottle of each treatment using an ocygen electrode, the other twelve bottles were closed and incubated at about 20 deg C in the dark.
- After 7, 14, 21, and 28 days, three bottles of each treatment were sacrificed for determination of the oxygen concentration.

CALCULATION OF RESULTS
- The oxygen demand in each test bottle after 7, 14, 21, and 28 days was calculated by subtracting the oxygen concentration measured at that time from that measured at the start of the test.
- The Biochemical Oxygen Demand (BOD) due to the test or control substances at each time was calculated (in mg O2/L) by subtracting the oxygen demand in the relevant inoculum control or other control bottle from that in the bottle under consideration; these crude values were then converted to values per mg substance.
- The degradation was expressed as BOD as percentage of the COD of the test substance.

Test substance : Asphalt, sulfonated, sodium salt, CAS Number 68201-32-1. XXXXXXXX XXXXX. Trade Name: Soltex Shale Inhibitor.

Reliability : (1) valid without restriction
Flag : Critical study for SIDS endpoint
30.01.2004 (13)

4.1 ACUTE/PROLONGED TOXICITY TO FISH

Type	:	semistatic
Species	:	other: Scophthalmus maximus
Exposure period	:	96 hour(s)
Unit	:	mg/l
Analytical monitoring	:	
LC50	:	= 1672
LC50 24h	:	> 1800
LC50 48h	:	> 1800
Method	:	OECD Guide-line 203 "Fish, Acute Toxicity Test"
Year	:	2002
GLP	:	yes
Test substance	:	other TS

Method : Based on OECD 203 and modified to marine conditions.

Result :

- 96-hour LC50 = 1672 mg/l (1194 to 2342 95% confidence limit interval)
- 24-hour and 48-hour LC50 > 1800 mg/l.
- The highest no-observed (lethal) effect concentration (NOEC) was estimated as 1000 mg/l.
- The lowest observed (lethal) effect concentration (LOEC) was 1800 mg/l.
- The lowest concentration giving 100% mortality could not be determined as there was only 57.1% mortality in the highest concentration.
- A mortality of 0% was observed in the control tank at the end of the test period.
- The test substance nominal concentrations were prepared as "water-accommodated fractions" according to the OSPAR guidelines, but significant amounts of test material remained suspended after the nominal settling period. The solution was filtered using a 63 um sieve to remove the suspended material and the subsequent filtrate used for the test. However, in test concentrations it was observed that some material sedimented out of solution at 24 hours onwards. This increased in quantity with increase in concentration and time.
- There was no chemical analytical confirmation of the actual dissolved concentrations. The dissolved concentrations were likely to be lower than the nominal concentrations as Soltex Additive was poorly soluble in water.

RAW DATA
- Cummulative percent mortality results are presented in the following format: Exposure period (hours) / control / 560 mg/l / 1000 mg/l / 1800 mg/l

0 / 0 / 0 / 0 / 0
24 / 0 / 0 / 0 / 0
48 / 0 / 0 / 0 / 14.3
72 / 0 / 0 / 0 / 57.1
96 / 0 / 0 / 0 / 57.1

Source : Phillips Petroleum Company, The Toxicity to Turbot (Scophthalmus maximus) of Soltex Additive - Report for Drilling Specialties Company. Study performed by Chemex Environmental International Limited, Cambridge, England for Drilling Specialties Company, Bartlesville, Oklahoma.

Test condition : TEST SPECIES
- Turbot (Scophthalmus maximus)
- Acclimation period: 6 June to 1 July 2002
- Acclimation conditions: Temperature: 13.5 to 14.5 deg C; Dissolved oxygen: >95% ASV
Mean length: 43.5 mm
Mean weight: 2.13 g

DILUTION WATER:
- The stocks of animals were maintained, and the tests performed, in standardised artificial seawater using Tropic Marin artificial sea salt.
- The measured salinity of the seawater used was 31 to 35 g/l sodium chloride.

TEST METHODS AND CONDITIONS
- A nominal 1000 mg/l solution of Soltex Additive was prepared in dilution water, shaken vigorously and allowed to stand for four hours.
- For each nominal concentration the required amount of homogenised sample was added to 12 litres of dilution water, mixed for 20-24 hours and then allowed to separate for four hours. The solution was filtered using a 63 um sieve, the filtrate was used for the test.
- A preliminary study had identified the 96 hour LC50 as being > 1000 mg/l and therefore definitive test concentrations were prepared as 0 (Control), 560, 1000, and 1800 mg/l.
- Volumes of 10 litres of test solution were prepared in aquaria. A control vessel of 10 litres dilution water was prepared.
- Seven turbot were placed in each of the test and control vessels.
- The pH value (to 0.1), dissolved oxygen (to 1% ASV), and temperature (to 0.5 deg C) were measured on each test and control solution immediately prior to initiating the test.
- The test and control solutions were replaced at 48 hours, the remaining live animals being transferred to freshly prepared test solutions.
- The test parameters were measured before and after each change of test solution, and observations of mortality were made daily.
- The test vessels were maintained at 15 +/- 1.5 deg C, with a light cycle of 16 hours light and 8 hours dark.

STATISTICS
- Cumulative mortalities were calculated for each test concentration and the control. LC50 values were estimated and 95% confidence limits calculated using ToxCalc version 5.0 "Comprehensive Toxicity Data Analysis and Database Software."

Test substance : Asphalt, sulfonated, sodium salt, CAS Number 68201-32-1 (XXXXXXXXX XXXXX). Trade Name: Soltex Additive.

Reliability : (2) valid with restrictions
Flag : Critical study for SIDS endpoint
30.01.2004 (2) (10) (14)

4.2 ACUTE TOXICITY TO AQUATIC INVERTEBRATES

Type	:	static
Species	:	Mysidopsis bahia (Crustacea)
Exposure period	:	96 hour(s)
Unit	:	mg/l
Analytical monitoring	:	no data
EC50	:	= 420000
Method	:	other: EPA 40 CFR Part 435
Year	:	1994

GLP : no data
Test substance : other TS

Result : 96-hour LC50 was 420,000 ppm (95% confidence interval of 368,000 ppm to 481,000 ppm).

The 96- hour LC50 for the standard reference toxicant (sodium lauryl sulfate) was 8.5 ppm, with a 95% confidence interval of 8.0 ppm to 9.1 ppm.

RAW DATA:
Results presented in the following format: Concentration (%) / Number exposed / Mortalities

0 / 20 / 0
1 / 20 / 0
3 / 20 / 0
5 / 20 / 0
10 / 20 / 0
25 / 20 / 0
50 / 20 / 15
100 / 20 / 20

Spearman-Karber Estimates:
- LC50: 42.04
- 95% lower confidence: 36.76
- 95% upper confidence: 48.08

Source : Phillips Petroleum Company, 96 Hour Range Finder Acute Toxicity Test of Drilling Fluid Suspended Particulate Phase - Based on Permit #: GMG290000. Study performed by Laboratory Technology, Inc., Kenner, Louisiana for Drilling Specialties Company, Bartlesville, Oklahoma.

Test condition : MATERIALS AND METHODS
- Based on those suggested by the EPA (40 CFR Part 435; 8/26/85).
- All equipment was washed with detergent, rinsed with tap water, acetone, deionized water, soaked in a 10% HCL bath, rinsed with tap water and finally deionized water.

Artificial Seawater Preparation: Made by mixing a commercial brand of synthetic sea salts (Hawaiian Marine Mix) with deionized water. The seawater was prepared at a salinity of 20 +/- 2 ppt and stored in a opaque drum with continuous aeration. Water was "seasoned" for several days before use and filtered through a 1.0 micron filter.

Organism Acquisition and Maintenance: Mysid shrimp (Mysidopsis bahia) were raised and maintained at 25 +/- 1 deg C and 20 +/- 2 ppt salinity. During maintenance and testing, mysids were fed approximately 50 brine shrimp nauplii per mysid daily. Test organisms were 4 to 6 days old.

TEST MEDIA PREPARATION:
- A one-half gallon sample of drilling fluid from Drilling Specialties Company was provided and stored at 4 deg C. The drilling fluid had a pH of 6.05 and did not emit a foul odor. The sample was thoroughly mixed for 30 minutes prior to use.
- The suspended particulate phase (SPP) was prepared by mixing the mud sample and artificial seawater in a 1 to 9 ratio in 2-L large-mouth Erlenmeyer flask.
- Mud/seawater slurry mixed for 5 minutes.
- pH of the slurry was measured and adjusted, if necessary, to within 0.2 units of the seawater by adding diluted hydrochloric acid while stirring.
- Slurry was allowed to settle for one hour.

- Supernatant (SPP) was decanted and SPP was mixed for another 5 minutes while the pH and dissolved oxygen were measured and adjusted if necessary.
- If the dissolved oxygen was less than 4.9 ppm, the SPP was aerated to at least 4.9 ppm which is 65 percent of saturation. Maximum aeration time was 5 minutes.
- The filterable and non-filterable residue of each SPP was measured according to the methods listed in the ASTM.

EXPERIMENTAL CONDITIONS
SPP test was conducted at 20 +/- 2 ppt salinity and 20 +/- 1 deg C. Dissolved oxygen, temperature, salinity, and pH were measured at 0, 24, 48, 72, and 96 hours.
- Test media was aerated during the entire test at an estimated rate of 50-144 cubic centimeters per minute.
- Light/dark cycle was maintained at 14L/10D during maintenance and testing.

EXPERIMENTAL PROCEDURE
- Nytex cups were used to confine the mysids in each test concentration. Cups were positioned in 8 inch Carolina Culture dishes which contained 1 liter of the test solution.
- 20 organisms were exposed to each test concentration of the prepared SPP, the control, and the standard reference toxicant.
- Organisms were selected, transferred and assigned to treatments, containers, and positions according to a modified randomization procedure as described in the EPA 40 CFR part 435.
- All live organisms were counted at 0, 24, 48, 72, and 96 hours in those dishes where turbidity and color did not preclude observation.

Test substance : Drilling fluid from Drilling Specialties Company containing Asphalt, sulfonated, sodium salt, CAS Number 68201-32-1. (XXXXXXXXXXXX).

Reliability : (2) valid with restrictions
Flag : Critical study for SIDS endpoint
30.01.2004 (8) (15)

Type : static
Species : other: Acanthomysis sculpta
Exposure period : 96 hour(s)
Unit : mg/l
Analytical monitoring :
EC50 Suspended Particulate Phase : = 205000
EC50 Liquid Phase : = 155000
Method : other: EPA Region 2 Drilling Mud Bioassay
Year : 1982
GLP : no data
Test substance : other TS

Method : Drilling Mud bioassay Test Procedures to be Employed Under EPA, Region 2, Offshore Exploratory Drilling Permits, Annexes I, II, and III. Procedures employed in bioassay testing were generally in accordance with those developed by the Mid-Atlantic Joint Industry Bioassay Program.

Result : The 96-hour LC50 values for Acanthomysis sculpta were 155,000 ppm in the Liquid Phase bioassay and 205,000 ppm for the Suspended Particulate Phase bioassay.

RAW DATA
Liquid Phase Bioassay:
- Number of Survivors -- Results are presented in the following format:

Test Medium Concentration (ppm v/v) / Replicate / 0 hr / 4 hr / 8 hr / 24 hr / 48 hr / 72 hr / 96 hr

1,000,000 / 1 / 10 / 10 / 7 / 4 / 0 / 0 / 0
1,000,000 / 2 / 10 / 10 / 8 / 3 / 0 / 0 / 0
1,000,000 / 3 / 10 / 9 / 9 / 2 / 1 / 0 / 0
1,000,000 / 4 / 10 / 10 / 9 / 4 / 0 / 0 / 0
1,000,000 / 5 / 10 / 10 / 7 / 4 / 0 / 0 / 0

500,000 / 1 / 10 / 10 / 10 / 7 / 0 / 0 / 0
500,000 / 2 / 10 / 10 / 10 / 9 / 0 / 0 / 0
500,000 / 3 / 10 / 10 / 10 / 9 / 0 / 0 / 0
500,000 / 4 / 10 / 10 / 10 / 6 / 0 / 0 / 0
500,000 / 5 / 10 / 10 / 10 / 8 / 8 / 0 / 0

200,000 / 1 / 10 / 10 / 10 / 10 / 9 / 8 / 7
200,000 / 2 / 10 / 10 / 10 / 8 / 8 / 6 / 5
200,000 / 3 / 10 / 10 / 10 / 8 / 8 / 6 / 5
200,000 / 4 / 10 / 10 / 10 / 9 / 9 / 5 / 4
200,000 / 5 / 10 / 10 / 10 / 10 / 7 / 3 / 3

100,000 / 1 / 10 / 10 / 10 / 9 / 9 / 9 / 9
100,000 / 2 / 10 / 10 / 10 / 8 / 8 / 7 / 7
100,000 / 3 / 10 / 10 / 10 / 10 / 10 / 10 / 9
100,000 / 4 / 10 / 10 / 10 / 10 / 10 / 9 / 9
100,000 / 5 / 10 / 10 / 10 / 9 / 9 / 8 / 8

50,000 / 1 / 10 / 10 / 10 / 10 / 10 / 10 / 10
50,000 / 2 / 10 / 10 / 10 / 10 / 9 / 8 / 8
50,000 / 3 / 10 / 10 / 10 / 10 / 10 / 10 / 10
50,000 / 4 / 10 / 10 / 10 / 9 / 9 / 8 / 8
50,000 / 5 / 10 / 10 / 10 / 10 / 10 / 10 / 10

0 (Control) / 1 / 10 / 10 / 10 / 10 / 10 / 9 / 9
0 (Control) / 2 / 10 / 10 / 10 / 10 / 10 / 10 / 10
0 (Control) / 3 / 10 / 10 / 10 / 10 / 10 / 10 / 10
0 (Control) / 4 / 10 / 10 / 10 / 9 / 9 / 9 / 9
0 (Control) / 5 / 10 / 10 / 10 / 10 / 10 / 10 / 10

Suspended Particulate Phase Bioassay:
- Number of Survivors results are presented in the following format: Test Medium Concentration (ppm v/v) / Replicate / 0 hr / 4 hr / 8 hr / 24 hr / 48 hr / 72 hr / 96 hr

1,000,000 / 1 / 10 / 10 / 9 / 7 / 0 / 0 / 0
1,000,000 / 2 / 10 / 10 / 9 / 8 / 1 / 0 / 0
1,000,000 / 3 / 10 / 10 / 10 / 7 / 0 / 0 / 0
1,000,000 / 4 / 10 / 10 / 9 / 6 / 0 / 0 / 0
1,000,000 / 5 / 10 / 10 / 8 / 7 / 0 / 0 / 0

500,000 / 1 / 10 / 10 / 10 / 7 / 4 / 2 / 2
500,000 / 2 / 10 / 10 / 10 / 7 / 7 / 4 / 4
500,000 / 3 / 10 / 10 / 10 / 8 / 6 / 4 / 3
500,000 / 4 / 10 / 10 / 10 / 7 / 6 / 4 / 2
500,000 / 5 / 10 / 10 / 10 / 5 / 5 / 5 / 2

200,000 / 1 / 10 / 10 / 10 / 10 / 9 / 7 / 7
200,000 / 2 / 10 / 10 / 10 / 9 / 8 / 8 / 8
200,000 / 3 / 10 / 10 / 10 / 9 / 8 / 8 / 8
200,000 / 4 / 10 / 10 / 10 / 7 / 7 / 7 / 6
200,000 / 5 / 10 / 10 / 10 / 8 / 8 / 4 / 4

100,000 / 1 / 10 / 10 / 10 / 9 / 9 / 9 / 9
100,000 / 2 / 10 / 10 / 10 / 7 / 7 / 6 / 6
100,000 / 3 / 10 / 10 / 10 / 10 / 10 / 7 / 7
100,000 / 4 / 10 / 10 / 10 / 9 / 9 / 8 / 8
100,000 / 5 / 10 / 10 / 10 / 10 / 10 / 10 / 9

50,000 / 1 / 10 / 10 / 10 / 10 / 10 / 10 / 10
50,000 / 2 / 10 / 10 / 10 / 10 / 10 / 10 / 10
50,000 / 3 / 10 / 10 / 10 / 8 / 8 / 7 / 7
50,000 / 4 / 10 / 10 / 10 / 8 / 8 / 8 / 8
50,000 / 5 / 10 / 10 / 10 / 9 / 9 / 9 / 8

0 (Control) / 1 / 10 / 10 / 10 / 10 / 10 / 9 / 9
0 (Control) / 2 / 10 / 10 / 10 / 10 / 10 / 10 / 10
0 (Control) / 3 / 10 / 10 / 10 / 10 / 10 / 10 / 10
0 (Control) / 4 / 10 / 10 / 10 / 9 / 9 / 9 / 9
0 (Control) / 5 / 10 / 10 / 10 / 10 / 10 / 10 / 10

Source : Phillips Petroleum Company, Drilling Mud Bioassay - Soltex - Acanthomysis sculpta and Macoma nasuta. Study performed by Marine Bioassay Laboratories, Watsonville, California for IMCO Services (Houston, Texas) and Drilling Specialties Company (Houston, Texas).

Test condition : LABORATORY FACILITIES
- Bioassay procedures conducted in MBL's marine laboratory located on the beach at Davenport Landing, California.
- Seawater system includes tandem intake lines extending 180 meters seaward from the beach and all cast-iron pumps delivering a flow of up to 2500 ppm each.
- Water is continuously supplied for use either unfiltered, sand-filtered, or sub-micron filtered, and can be heated or cooled to within 0.3 deg C of the desired temperature.
- 14-hour light/ 10-hour dark photoperiod during animal acclimation and testing periods.
- Test containers were wide-mouth glass jars (3.78 liters) containing 2 liters of test material.

TEST ORGANISMS
- Acanthomysis sculpta were collected by MBL personnel from kelp beds near Monterey, California and transported in aerated plastic buckets.
- Mysids were held for acclimation to test temperature and Davenport seawater for at least two days prior to testing.
- During acclimation and testing, mysids were fed brine shrimp nauplii.

TEST MATERIAL SAMPLING AND PREPARATION
- The drilling mud to be bioassayed was prepared and packed according to Region 2 procedures. Samples were stored at 2-4 deg C until preparation began.
- After preliminary pH testing and inspection, 22.7 liters of composited sample were transferred to a clean 190 liter polyethylene barrel and 90.8 liters of Davenport seawater were added.
- The pH of the resulting mud-seawater slurry was checked and found to be within 0.1 pH unit of ambient seawater.
- The mud-seawater slurry was mixed by vigorous aeration for 30 minutes.
- Following a one hour settling period the resulting elutriate (which required no centrifugation) was siphoned into clean buckets.
- The remaining sediment was reserved for use as the Solid Phase bioassay test material.
- Half the elutriate was filtered through a pre-washed 0.5 uM cartridge type acetate filter and retained as Liquid Phase test material. The unfiltered elutriate comprised the Suspended Particulate Phase test material.

BIOASSAY TEST PROCEDURES
- Liquid and Suspended Particulate Phase bioassays were conducted concurrently.
- Two liters of freshly prepared and appropriately diluted test material was added to each jar.
- Five replicates of five test concentrations and of control seawater were established and ten animals were used for each replicate.
- Survivors of the original ten animals per jar were recorded as test data at 4, 8, 24, 48, 72, and 96 hours after testing began.
- Dead animal were removed.
- Dissolved oxygen, temperature, salinity and pH were measured in each test container once daily.
- Mysids were fed once each day with 50-100 Artemia nauplii per mysid.

DATA ANALYSIS
- In order to facilitate calculation, LC50 values were obtained by computer regression analysis, modified to accommodate the probit (mortality) and logrithmic (elutriate concentration) scaled axes.

Test substance : Drilling mud from Drilling Specialties Company containing Asphalt, sulfonated, sodium salt, CAS Number 68201-32-1. (XXXXXXXXXXX). Trade Name: Soltex.

Reliability : (2) valid with restrictions
Flag : Critical study for SIDS endpoint
30.01.2004 (9)

4.3 TOXICITY TO AQUATIC PLANTS E.G. ALGAE

Species	:	Skeletonema costatum (Algae)
Endpoint	:	growth rate
Exposure period	:	95 hour(s)
Unit	:	g/l
Analytical monitoring	:	
NOEC	:	= 1
EC50	:	= 4
EC10	:	= .5
EC90	:	= 29.5
Method	:	other: ISO/DIS 10253
Year	:	1991
GLP	:	yes
Test substance	:	other TS

Result : The EC50 with respect to inoculum viability followed by logistic growth (EeC50) was found to be 4.0 g/L (95% confidence interval of 2.2 - 7.3 g/L). The corresponding EeC10 and EeC90 values were 0.5 and 29.5 g/L respectively.

The EC50 with respect to the area under the growth curve (EbC50) was found to be 8.3 g/L (in the range 3.2 - 10.0 g/L). The corresponding EbC10 and EbC90 values were 0.7 g/L (in the range 0.3 - 1.0 g/L) and > 10.0 g/L respectively.

The no-observed-effect-concentration (NOEC) was estimated to be 1.0 g/L.

The EbC and the EeC values were in the same range confirming the biomass dependent nature of the effects of the test substance.

Results were based on nominal concentrations of 1% Soltex Solution.

Microscopic examination of the cells at the start and end of the incubation period revealed no abnormalities; the chainlength (number of cells per particle) of the algal particles was, however, low in all cultures except that exposed to 3.28 g/L test substance.

RAW DATA
- Results of the model calculations for effect on inoculum viability followed by logistic growth (EeC50) -- Calculated Values (1E+3 particles/ml) where results are presented in the following format: Time (h) / 0 g/L 1% Soltex / 0.3 g/L 1% Soltex / 1.0 g/L 1% Soltex / 1.8 g/L 1% Soltex / 3.3 g/L 1% Soltex / 10 g/L 1% Soltex

0.0 hr / 0.7 / 0.7 / 0.6 / 0.5 / 0.4 / 0.2
22.5 hr / 6.6 / 6.2 / 5.4 / 4.6 / 3.7 / 1.8
47.0 hr / 61.2 / 58.3 / 51.4 / 45.4 / 37.1 / 19.3
68.0 hr / 213.0 / 208.4 / 196.4 / 184.6 / 165.7 / 108.8

- Mean area under the growth curve (A)
0 g/L 1% Soltex = 14332
0 g/L 1% Soltex = 13078
0.3 g/L 1% Soltex = 13307
1.0 g/L 1% Soltex = 11843
1.8 g/L 1% Soltex = 10932
3.3 g/L 1% Soltex = 9688
10 g/L 1% Soltex = 6308

- Percentage reduction in growth (IA)
0 g/L 1% Soltex = 0%
0 g/L 1% Soltex = 0%
0.3 g/L 1% Soltex = 3%
1.0 g/L 1% Soltex = 14%
1.8 g/L 1% Soltex = 20%
3.3 g/L 1% Soltex = 29%
10 g/L 1% Soltex = 54%

Source : Phillips Petroleum Company, Effect of a 1% Soltex Solution (262-100-3) on the Growth of the Marine Alga Skeletonema costatum (ISO/DIS 10253). Study performed by TNO Environmental and Energy Research, Delft, The Netherlands for Drilling Specialties Company, Bartlesville, Oklahoma.

Test condition : TEST SUBSTANCE:
- 1% Soltex Solution (262-100-3), a black liquid.
- Sample was stored at room temperature.
- Sample was stated to be soluble in water
- Sample prepared by sponsor as follows: "Using the Soxhlet Extraction procedure, dissolved 1.75 g Soltex in 175 ml tap water. The insoluble portion of Soltex was removed from the extraction thimble, and it was added to the Soltex solution."

TEST ORGANISM
- Marine alga Skeletonema costatum (ISTPM P4).
- A preculture of algae in the exponential growth phase was prepared as detailed in ISO/DIS 10253.

TEST MEDIUM
- Prepared in natural seawater with a salinity of approximately 32% and sterilized by micropore filtration.
- Stock solution 1: 3.2 mg/L K3PO4.H2O and 50.0 mg/L NaNO3
- Stock solution 2: 14.9 mg/L Na2SiO3.9H2O
- Stock solution 3: 140.0 ug/L C6H8O7Fe.3H2O; 605.0 ug/L MnCl2.4H2O; 150.0 ug/L ZnSO4.7H2O; 0.6 ug/L CuSO4.5H2O; 1.5 ug/L CoCl2.6H2O; 17.1 mg/L H3BO3; and 15.0 mg/L Na2EDTA.

- Stock solution 4: 25 ug/L Thiamin hydrochloride, 0.005 ug/L Biotin, and 0.05 ug/L B12.
- The medium as prepared by making 1 ml of stock solution 1, 0.52 ml of stock solution 2, 10 ml of stock solution 3, and 1 ml of stock solution 4 up to one litre with natural seawater.
- The pH was 8.0 +/- 0.2 after equilibration.

PREPARATION OF TEST SOLUTIONS
- Stock solutions prepared by dissolving 10, 97.6, and 1009.0 mg respectively in 1000 ml of algal medium (range-finding test) or 0.150, 0.52, 0.91, 1.64, and 5.01 g respectively in 500 ml of algal medium (growth inhibition test).
- The stock solutions for the range-finding test were used directly in the test.
- From the stock solutions prepared for the growth inhibition test, appropriate dilutions were prepared in algal medium to yield final concentrations of 0, 0.30, 1.0, 1.8, 3.3, and 10.0 g/L.

RANGE-FINDING TEST
- 2.6 ml of algal preculture containing 7.6E+4 particles/ml was added to a hundred ml of the appropriate solutions of the test substance and yielded a mean measured inoculum particle density in the control cultures of 2.2E+3 particles/ml.
- Test carried out in duplicate with two controls with algae only and a single background series containing test substance without algae.
- All flasks were incubated at 20 +/- 1 deg C and shaken (100 rpm) in an orbital shaker.
- Light intensity radiated by the fluorescent lamps was within the standard range of 60-120 umol/s/m2.
- After 3 days of incubation one sample was taken from each flask, and the number of particles per ml in the samples was determined with the aid of a Coulter Counter model TAII.

GROWTH INHIBITION TEST
- Test flasks, test solutions, and algal medium were prepared as detailed above.
- A suspension of algae in the algal medium containing 1E+5 cells/ml was prepared by dilution of a preculture containing 3.1E+5 particles/ml.
- Addition of 1.0 ml of this algal suspension to 100 ml of the appropriate solutions of the test substance in the test flasks yielded a mean measured inoculum particle density in the control cultures of 0.8E+3 particles/ml.
- All flasks were incubated at 20 +/- 1 deg C and shaken (100 rpm) in an orbital shaker.
- One sample was taken from each flask after 0, 22.5, 47, 68, and 95 hours, and the number of algal cells per ml in the samples was determined with the aid of a Coulter Counter model TAII.
- pH was measured at the start (medium without algae) and after 68 and 95 h in selected cultures. The pH of the algal medium at the start of the test was 7.8. The pH of the medium containing different test substance concentrations remained constant (pH 8.0 - 8.1) during the test. In the presence of algae, however, the pH was found to increase with algal cell density to pH 8.7 - 9.1 after 3 days and to pH 8.4 - 8.7 after 4 days.
- The morphology of the algae was examined visually with the aid of a microscope at the start and at the end of the test.

CALCULATION OF EC VALUES
- Algal particle density was obtained by subtraction of the number of particles in the background control series (without algae) from thenumber of particles in the test series. The mean values calculated were used for further calculations.
- The effect of a test substance on the growth of algae is expressed by quantities denoted as EC10, EC50, or EC90, i.e., the concentration of test

substance that reduced the growth rate, the yield or the viability of the inoculum cells by 10%, 50%, or 90% respectively.
- EC values with respect to the inoculum viability followed by logistic growth (EeC values), assuming an error proportional to the number of particles, were calculated by means of a parametric model developed by Kooijman et al. (1983). The values obtained in the last sampling period (95h) were omitted for model calculations because the S. costatum cell chains had broken, resulting in irregular particle counts.
- EC values with respect to the area under the growth curve (EbC values) were calculated by the method given in ISO/DIS 10253. The values were calculated by a linear interpolation of a plot of the percentage reduction in growth (IA) against the log concentration of the test substance.

DETERMINATION OF THE NOEC
- The "no-observed-effect-concentration" was estimated by visual comparison of both the measured and calculated growth curves of the treated algal suspensions with those of the controls.

Test substance : 1% Soltex Aqueous Solution (262-100-3). Asphalt, sulfonated, sodium salt, CAS Number 68201-32-1. XXXXXXXXXXX. Trade Name: Soltex
Reliability : (1) valid without restriction
Flag : Critical study for SIDS endpoint
30.01.2004 (6) (7) (11)

4.5.2 CHRONIC TOXICITY TO AQUATIC INVERTEBRATES

Species : other aquatic mollusc: Macoma nasuta
Endpoint : mortality
Exposure period : 10 day
Unit : mg/l
Analytical monitoring :
Method : other: EPA Region 2 Drilling Mud Bioassay
Year : 1982
GLP : no data
Test substance : other TS

Method : Drilling Mud bioassay Test Procedures to be Employed Under EPA, Region 2, Offshore Exploratory Drilling Permits, Annexes I, II, and III. Procedures employed in bioassay testing were generally in accordance with those developed by the Mid-Atlantic Joint Industry Bioassay Program.

Result : There was only a single mortality of Macoma nasuta in five experimental tanks (mean percent survival = 99%). This result was not statistically different compared to its control and it was concluded that SOLTEX drilling mud is not lethally toxic to Macoma nasuta.

RAW DATA
Results are presented in the following format: Replicate / Percent Survival at Day 10 in Control / Percent Survival at Day 10 in Soltex

1 / 100% / 95%
2 / 100% / 100%
3 / 100% / 100%
4 / 100% / 100%
5 / 100% / 100%

Mean / 100% / 99%
Variance (s2) / 0.0 / 5.0

Source : Phillips Petroleum Company, Drilling Mud Bioassay - Soltex -

Acanthomysis sculpta and Macoma nasuta. Study performed by Marine Bioassay Laboratories, Watsonville, California for IMCO Services (Houston, Texas) and Drilling Specialties Company (Houston, Texas).

Test condition : LABORATORY FACILITIES
- Bioassay procedures conducted in MBL's marine laboratory located on the beach at Davenport Landing, California.
- Seawater system includes tandem intake lines extending 180 meters seaward from the beach and all cast-iron pumps delivering a flow of up to 2500 gpm each.
- Water is continuously supplied for use either unfiltered, sand-filtered, or sub-micron filtered, and can be heated or cooled to within 0.3 deg C of the desired temperature.
- 14-hour light/ 10-hour dark photoperiod during animal acclimation and testing periods.
- Test containers used for solid phase bioassays are all-glass aquaria of 30 liters capacity with 1000 cm2 bottom area.

TEST ORGANISMS
- Macoma nasuta were collected from Tomales Bay. Clams were held in control sediment at ambient seawater temperature (13-15 deg C).
- At least 5 days prior to testing, the required number of animals were withdrawn from the holding tanks, placed in experimental aquaria with control sediment, and the temperature adjusted to 15 deg C.
- During holding, acclimation, and testing, the clams fed on phytoplankton and detritus present in Davenport seawater system; no additional food was provided.

TEST MATERIAL SAMPLING AND PREPARATION
- The drilling mud to be bioassayed was prepared and packed according to Region 2 procedures. Samples were stored at 2-4 deg C until preparation began.
- After preliminary pH testing and inspection, 22.7 liters of composited sample were transferred to a clean 190 liter polyethylene barrel and 90.8 liters of Davenport seawater were added.
- The pH of the resulting mud-seawater slurry was checked and found to be within 0.1 pH unit of ambient seawater.
- The mud-seawater slurry was mixed by vigorous aeration for 30 minutes.
- Following a one hour settling period the resulting elutriate (which required no centrifugation) was siphoned into clean buckets.
- The remaining sediment was reserved for use as the Solid Phase bioassay test material.

BIOASSAY TEST PROCEDURES
- Five replicates of sample and control treatments.
- A 3 cm layer of control mud was added to each tank, the tanks filled with water, and 20 Macoma nasuta added to each tank.
- After 48 hours of acclimation to the laboratory test environment, 1.5 cm of drilling mud was added to each sample treatment tank and an additional 1.5 cm layer of control mud was added to each control tank.
- A one hour settling period was allowed after sample addition, after which the flow-through seawater system was turned on.
- Solid phase bioassays continued for 10 days. At least twice each day, laboratory environmental control systems were checked for continuity.
- Daily measurements were made of system salinity and temperature and of the dissolved oxygen level in each aquarium.
- After the 10 day bioassay period, the contents of each tank were washed through a 3 mm plastic screen with seawater and the animals were retreived and counted.
- Test data were the number of survivors.

DATA ANALYSIS

- Solid Phase bioassays were analyzed by either Analysis of Variance or its non-parametric analogue for 2-sample comparison, the Mann-Whitney test.
- Variance homogeneity was the criteria which determined the appropriate analytical series (parametric or non-parametric).
- In all statistical tests, significance was based upon an alpha-level of 0.05.

Test substance : Drilling mud from Drilling Specialties Company containing Asphalt, sulfonated, sodium salt, CAS Number 68201-32-1 (XXXXXXXXXXX). Trade Name: Soltex.

Reliability : (1) valid without restriction
30.01.2004 (9)

5.1.1 ACUTE ORAL TOXICITY

Type	:	LD50
Species	:	rat
Strain	:	Sprague-Dawley
Sex	:	male/female
Number of animals	:	20
Vehicle	:	water
Value	:	> 5000 mg/kg bw
Method	:	EPA OPP 81-1
Year	:	1985
GLP	:	yes
Test substance	:	other TS

Result : LC50 was estimated to be greater than 5000 mg/kg bw in both male and female rats.

No animals were found dead during either the Dose Range or Single Dose Studies.

Clinical signs noted during the Dose Range Study were limited to instances of soft feces and/or a rough coat in all groups at one or more intervals.

Clinical signs noted in the Single Dose Study consisted of soft feces in three males and all females at one hour post dose, in all animals at two and four hours, and a rough coat in all animals on Day 1. All animals appeared normal from Day 2 through termination. All animals gained weight from initiation to termination.

Gross pathology findings noted in animals on the Dose Range Study were limited to pale adrenals in Groups 1-3 (1000 mg/kg, 2000 mg/kg, and 3000 mg/kg) and Group 5 (5000 mg/kg) males and in Group 2 (2000 mg/kg) and Groups 4-5 (4000 mg/kg and 5000 mg/kg) females and dark adrenals were noted in the Group 4 (4000 mg/kg) male. No observable gross pathology was noted in any of the Single Dose Study animals upon necropsy.

Source : Phillips Petroleum Company Acute Oral Toxicity Study in Rats - Product #2 - Final Report. Study performed by Hazleton Laboratories America Inc., Vienna Virginia for Drilling Specialties Company Bartlesville, Oklahoma.

Test condition : Test Animals:
- Young adult male and female albino rats (weighing between 200-300 grams) of the Sprague-Dawley strain.
- Maintained individually in elevated wire-mesh cages in temperature-controlled and humidity monitored quarters.
- Acclimation period of approximately one week.
- 12-hour light/dark cycle.

Methods:
- For Dose Range Study, one rat/sex was dosed at levels of 1000, 2000, 3000, 4000 and 5000 mg/kg bw (initial body weights of males ranged from 205.7 to 221.8 g, and the initial body weights of females ranged from 203.0 to 236.1 g).
- Five rats/sex were assigned to the Single Dose Study and were dosed at a level of 5000 mg/kg bw (initial body weights of the males ranged from 252.4 to 299.0 g, and the initial body weights of the females ranged from 227.6 to 264.7 g).
- The dosage factor was 20 ml/kg.

Preparation and Administration of Test Material:
- Distilled water was added to the test sample to bring it up to the desired volume.
- All mixtures were stirred during dosing.

Observations:
- Dose Range Study: each animal was observed for signs of toxic and pharmacologic effects at 1, 2, 4, 24, and 48 hours after test material administration.
- Single Dose Study: each animal was observed for signs of toxic and pharmacologic effects at 1, 2, and 4 hours after test material administration and once daily thereafter to 14 days.
- Mortality/moribundity was recorded twice daily.
- Individual body weights were recorded immediately prior to treatment and at termination in both studies and at Day 7 in the Single Dose Study.
- At the end of the study an acute oral LD50 was estimated for each sex.

Pathology: At termination of the Dose Range and Single Dose Studies, all rats were sacrificed by carbon dioxide asphyxiation and necropsied. Observations were recorded.

Test substance : Asphalt, sulfonated, sodium salt, CAS Number 68201-32-1. XXXXXXXXX purity. Trade Name: Soltex.

Reliability : (1) valid without restriction
Flag : Critical study for SIDS endpoint
30.01.2004 (4)

Type : LD50
Species : rat
Strain : Sprague-Dawley
Sex : male/female
Number of animals : 20
Vehicle : water
Value : > 5000 mg/kg bw
Method : EPA OPP 81-1
Year : 1985
GLP : yes
Test substance : other TS

Result : LD50 was estimated to be greater than 5000 mg/kg bw in both male and female rats.

No animals were found dead during either the Dose Range or Single Dose Studies.

Clinical signs noted during the Dose Range Study were limited to instances of soft feces and/or a rough coat in all groups. All rats appeared normal at termination.

Clinical signs were noted among all animals in the Single Dose Study and consisted of soft feces, a rough coat and/or red stains on the nose and/or eyes at one or more intervals during the study. All animals appeared normal from Day 3 through termination. All animals gained weight from initiation to termination.

Gross pathology findings noted in animals on the Dose Range Study were limited to bright red lungs in the group 4 (4000 mg/kg) female and pale adrenals in the Group 5 (5000 mg/kg) female. No observable gross pathology was noted in any of the Single Dose Study animals upon necropsy.

Source : Phillips Petroleum Company Acute Oral Toxicity Study in Rats - Product #5 - Final Report. Study performed by Hazleton Laboratories America Inc., Vienna Virginia for Drilling specialties Company Bartlesville, Oklahoma.

Test condition : Test Animals:
- Young adult male and female albino rats (weighing between 200-300 grams) of the Sprague-Dawley strain.
- Maintained individually in elevated wire-mesh cages in temperature-controlled and humidity monitored quarters.
- Acclimation period of approximately one week.
- 12-hour light/dark cycle.

Methods:
- For Dose Range Study, one rat/sex was dosed at levels of 1000, 2000, 3000, 4000 and 5000 mg/kg bw (initial body weights of males ranged from 209.0 to 233.8 g, and the initial body weights of females ranged from 222.4 to 232.1 g).
- Five rats/sex were assigned to the Single Dose Study and were dosed at a level of 5000 mg/kg bw (initial body weights of the males ranged from 290.6 to 300.8 g, and the initial body weights of the females ranged from 237.1 to 258.8 g).
- The dosage factor was 20 ml/kg.

Preparation and Administration of Test Material:
- Distilled water was added to the test sample to bring it up to the desired volume.
- All mixtures were stirred during dosing.

Observations:
- Dose Range Study: each animal was observed for signs of toxic and pharmacologic effects at 1, 2, 4, 24, and 48 hours after test material administration.
- Single Dose Study: each animal was observed for signs of toxic and pharmacologic effects at 1, 2, and 4 hours after test material administration and once daily thereafter to 14 days.
- Mortality/moribundity was recorded twice daily.
- Individual body weights were recorded immediately prior to treatment and at termination in both studies and at Day 7 in the Single Dose Study.
- At the end of the study an acute oral LD50 was estimated for each sex.

Pathology: At termination of the Dose Range and Single Dose Studies, all rats were sacrificed by carbon dioxide asphyxiation and necropsied. Observations were recorded.

Test substance : Asphalt, sulfonated, sodium salt, CAS Number 68201-32-1. XXXXXXXX purity. Trade Name: Soltex 31.

Reliability : (1) valid without restriction
Flag : Critical study for SIDS endpoint
15.01.2004

(5)

5.1.2 ACUTE INHALATION TOXICITY

5.1.3 ACUTE DERMAL TOXICITY

5.1.4 ACUTE TOXICITY, OTHER ROUTES

5.4 REPEATED DOSE TOXICITY

5.5 GENETIC TOXICITY 'IN VITRO'

5.6 GENETIC TOXICITY 'IN VITRO'

5.8 TOXICITY TO REPRODUCTION

5.9 DEVELOPMENTAL TOXICITY/TERATOGENICITY

(1) BCO Analytical Services B.V, 1997. Determination of the Partition Coefficient (n-octanol/water), HPLC method, of Soltex Shale Inhibitor, BCO project number 3096110462, Breda, The Netherlands.

(2) Chemex Environmental International Limited. 2002. The Toxicity to Turbot (Scophthalmus maximus) of Soltex Additive - Report for Drilling Specialties Company. Chemex reference ENV6103/050221. Cambridge, England.

(3) Chemex Environmental International Limited. 2003. The Bioaccumulation Potential of Sulphonated Asphalt Additive - Report for Drilling Specialties Company. Chemex reference ENV6222/100202. Cambridge, England.

(4) Hazleton Laboratories America, Inc. 1985. Acute Oral Toxicity Study in Rats - Product #2, Final Report. Project No. 2375-101. Vienna, Virginia.

(5) Hazleton Laboratories America, Inc. 1985. Acute Oral Toxicity Study in Rats - Product #5, Final Report. Project No. 2375-104. Vienna, Virginia.

(6) ISO/DIS 10253: Water quality. Marine algal growth inhibition test with Skeletonema costatum and Phaeodactylum tricornutum. Document no.: ISO/TC 147/SC5/WG5/N120. Nederlands Normalisatie-instituut, Delft (March 1991).

(7) Kooijman, S.A.L.M., A.O. Hanstveit, and H. Oldersma. 1983. Parametric Analysis of Population Growth in Bioassays. Water Research 17, pp. 527-538.

(8) Laboratory Technology, Inc. 1994. 96 Hour Range Finder Acute Toxicity Test of Drilling Fluid Suspended Particulate Phase - Based on Permit #: GMG290000. Lab Technology Control# DS019. Kenner, Louisiana.

(9) Marine Bioassy Laboratories. 1982. Drilling Mud Bioassay - Soltex - Acanthomysis sculpta and Macoma masuta - Prepared for IMCO Services (Houston Texas) and Drilling Specialties Company (Houston, Texas). Watsonville, California.

(10) OSPAR / PARCOM. 1995. Protocols on Methods for the Testing of Chemicals Used in the Offshore Industry.

(11) TNO Environmental and Energy Research. 1991. Effect of a 1% Soltex Solution (262-100-3) on the Growth of the Marine Alga Skeletonema costatum (ISO/DIS 10253), TNO Study no. IMW-91-0072-01. Delft, The Netherlands.

(12) TNO Environmental and Energy Research. 1991. The Biodegradability of the Product 3.5% Bentonite Slurry with 1% Soltex (262:100-2) in Seawater According to a Proposed EC Test Guideline (Closed Bottle Test), TNO Study no. IMW-91-0073-01. Delft, The Netherlands.

(13) TNO Environmental and Energy Research. 1991. The Biodegradability of the Product Soltex Shale Inhibitor in Seawater According to a Proposed EC Test Guideline (Closed Bottle Test), TNO Study no. IMW-91-0021-01. Delft, The Netherlands.

(14) ToxCalc Version 5.0 "Comprehensive Toxicity Data Analysis and Database Software," copyright 1994-1996.

(15) U.S. Environmental Protection Agency, 1993. Industrial Technology Division, March 4, 1993. Appendix 2 to Subpart A of Part 435 - Drilling Fluids Toxicity Test Regulation for offshore subcategory of the oil and gas extraction point source category, Federal Register Vol. 58, No. 41, 12507-12512 / Thursday, March 4, 1993 (40 CFR Part 435).

(16) United States Environmental Protection Agency, Office of Pollution Prevention and Toxics and Syracuse Research Corporation, 2000. EPI Suite v 3.10 (April, 2001).